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NAVAL AIR WARFARE CENTER AIRCRAFT DIVISION
PATUXENT RIVER, MARYLAND



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REPORT NO: NAWCADPAX/TR-2002/243

CORROSION PREVENTIVE COMPOUNDS FOR CORROSION PREVENTION/MITIGATION (AERMET 100 STEEL)

by

**Dr. Eun U. Lee
Mr. Henry C. Sanders**

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14. ABSTRACT The role of corrosion preventive compound (CPC) in corrosion, specifically general corrosion and a localized corrosion: stress corrosion cracking (SCC) and hydrogen embrittlement of AerMet 100 steel was investigated in 3.5% NaCl solution. Eighteen CPCs were evaluated for the effectiveness and persistency in corrosion prevention/mitigation, determining the essential parameters: open circuit potential, effective period, and threshold stress intensity for stress corrosion cracking (K_{ISCC}). It was found that: (1) CPC ZC-010 is most effective and persistent in prevention/mitigation of general corrosion, respectively, (2) Carwell AR500 and Omega 2775 are best in that of SCC, and (3) Break Free SMX is optimum in that of hydrogen embrittlement.				
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SUMMARY

AerMet 100 steel specimens, bare and coated with 18 different corrosion preventive compounds (CPCs), were subjected to open circuit potential (OCP) measurement and stress corrosion cracking (SCC) test, employing rising step load method, in 3.5% NaCl solution. The CPC coating improved the resistance to general corrosion, measured with OCP, by 1.4 ~ 28.3 times and they were effective for 6 ~ 24 hr. It also improved the SCC resistance, measured with K_{ISCC} at OCP, by 3.6 ~ 4.5 times and the hydrogen embrittlement resistance, measured with K_{ISCC} at electrode potential $V_{SCE} = -1.2$ V, by 2.4 ~ 6.5 times. Among the 18 tested CPCs, CPC ZC-010 is most effective and persistent in prevention/mitigation of general corrosion. Carwell AR500 and Omega 2775 are best in that of SCC and Break Free SMX optimum in that of hydrogen embrittlement.

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INTRODUCTION

Many metallic components of aircraft are susceptible to corrosion, including general corrosion and localized corrosion, such as stress corrosion cracking (SCC), corrosion fatigue, pitting, etc., and hydrogen embrittlement. Such a material degradation by corrosion or hydrogen embrittlement threatens the aircraft safety, adversely affects the force readiness, and increases the maintenance burden. The yearly Navy corrosion cost amounts to \$1 billion and increases as the aircraft ages.

Extensive studies have been conducted to establish the means of preventing, retarding, and controlling corrosion, extending service life and saving life cycle cost. One of them is the use of corrosion preventive compound (CPC), growing among aircraft manufacturers and maintainers. Many CPCs develop a layer of surface film that protects the metal from corrosion by reducing the transport rate of corrosive species. Some others remove the corrosive environment from the metal and are called "water displacing CPCs." They also have strong penetrant properties to allow transportation of the fluid into joints and narrow crevices. Those CPCs are classified as (1) passivators, (2) organic CPCs, including slushing ones and pickling ones, and (3) vapor-phase ones. Passivators are usually inorganic oxidizing substances (e.g., chromates, nitrites, or molybdates) that passivate the metal and shift the corrosion potential several tenths volt in the noble direction. Nonpassivating CPCs, such as the pickling ones, are usually organic substances that have only a slight effect on the corrosion potential, changing it either in the noble or active direction usually not more than a few milli- or centivolts. In general, the passivating CPCs reduce corrosion rates to very low values, being more efficient in this regard than most of the nonpassivating types. They represent, therefore, the better CPCs available for certain metal-environment combinations. Commercial CPCs are normally mixtures of several classes of CPCs, some of which function as anodic inhibitors and others as cathodic inhibitors. Experience has shown that such mixtures often are more effective than CPCs used separately, evidencing "synergism" (reference 1). However, their effectiveness and persistency have not been fully established for aircraft components.

Some of the major studies on CPCs, reported in literature, are as follows. O'Neill and Smith (reference 2) and Machin and Mann (reference 3) compared the fatigue lives of double-lap joints with and without CPC in various environments and concluded that the addition of a CPC to a joint often reduced the fatigue life of the joint. Parrish et al. (references 4 and 5) and Lynch et al. (reference 6) reported the retardation of SCC, increasing K_{ISCC} and decreasing SCC velocity, for D6AC high strength low alloy steel in aqueous media by oxidizing inhibitors. Mousley (reference 7) observed acceleration of fatigue cracking by penetrating fluids for corrosion prevention in riveted joints of Alclad 2024-T3 alloy sheets. Verink and Das (reference 8) screened over 200 potential CPCs to test their effectiveness of controlling hydrogen embrittlement and SCC of 4340, 300M, 17-4 PH, HP 9-4-30, and HY 180 steels. The three final selected CPCs were the "blocking type" ones (piperidine, piperazine, and Nalco 39L), which reduced the hydrogen content at the fracture surface and the SCC rate and increased the K_{ISCC} . According to Agarwala (references 9 and 10), CPCs retarded the corrosion fatigue crack growth in 4340 steel exposed to moist air and chloride-containing environment and the SCC of 7075-T6

aluminum alloy in chloride-containing environment. Kim et al. (reference 11) investigated various corrosion inhibitors, such as $\text{Na}_2\text{Cr}_2\text{O}_7$, Na_2CrO_4 , Na_2MoO_4 , and NaNO_2 , for their effect on the corrosion-wear process of AISI M50 and 52100 steels in NaCl environments. They found that the corrosion inhibitors effectively passivated the steels and decreased the corrosion activities. Trabanelli et al. (reference 12) studied the possibility of inhibiting or delaying SCC of AISI 304 stainless steel wires in 1 M HCl solutions by using organic additives. Some of the organic substances, such as phenylthiourea, benzimidazole-2-thiol, and benzothiazole-2-thiol, were found to inhibit the SCC. Trabanelli et al. (reference 13) also tested different types of organic additives (N- and S-containing and an acetylenic derivative) as inhibitors for the intergranular corrosion (IGC) of a sensitized AISI 304 stainless steel in 1 N sulfamic acid solution at 70°C. The only organic substances that could inhibit the IGC were those molecules containing a sulfur atom with one unshared lone electron pair. Shoji et al. (reference 14) found n-hexylamine very effective for the retardation of SCC of A533B CL.1 and 304 steels in high-temperature water. It has been shown by Hinton et al. (reference 15) that the velocity of SCC was significantly reduced by adding a small amount of CPC to an already corrosive environment. Khobaid (reference 16) showed that addition of a CPC to a corrosive environment reduced or removed the deleterious effects of the environment on fatigue crack growth in aluminum. Russo et al. (reference 17) investigated the influence of CPC on the fatigue life of aluminum one and a half dog-bone specimens. At low applied stresses, the fatigue life was reduced, but the reduction was only marginal at higher applied stresses. The addition of CPC reduced the amount of fretting and changed the failure mode. The work done by Shankar et al. (reference 18) indicates that CPCs have either a beneficial or detrimental effect on the life of a single fastener double-lap-joint, depending on the ratio of the applied force to the clamping force. The addition of CPC caused a variation in the failure mechanism and the location of crack initiation. Purry et al. (reference 19) conducted fatigue tests of a 2024-T351 aluminum alloy in air, distilled water, and CPC. They found the fatigue crack growth rate in CPC greater than that in distilled water.

Advanced aircraft landing gear and other fracture critical components demand strong and tough materials to achieve higher performance and greater reliability. In the past, 300M and AF 1410 steels were accepted as the materials for aircraft landing gear because of the high strength (300M) and the great fracture toughness (AF 1410). In 1990, a new Co-Ni alloy steel, strengthened with C, Cr, and Mo, AerMet 100, was developed. This steel has an outstanding combination of high ultimate tensile strength (UTS), 280 – 300 ksi, and high plane strain fracture toughness (K_{IC}), exceeding 100 $\text{ksi}\sqrt{\text{in}}$. The UTS and K_{IC} are similar to and much greater than those of 300M steel, 290 ksi and 59 $\text{ksi}\sqrt{\text{in}}$, respectively. On the other hand, they are greater and less than those of AF 1410 steel, 230 – 250 ksi and 150 $\text{ksi}\sqrt{\text{in}}$, respectively. Therefore, AerMet 100 steel can replace 300M steel to increase damage tolerance and AF 1410 steel to achieve weight reduction. This suggests that AerMet 100 steel has a great potential for application to fracture critical components. A typical example is its use as the material for F/A-18E/F aircraft landing gear, wingfold transmission, wing pivot pin, wheel bolts, etc. Therefore, AerMet 100 steel is an alloy of interest and importance to the Navy. However, this steel is susceptible to general and localized corrosion (references 20 and 21) and requires protection against such corrosion. As one of the protection means, CPC application is considered. However, its effectiveness and persistency remain to be determined.

With the above background knowledge, this study was initiated to test, evaluate, and compare some currently available CPCs with respect to the effectiveness and persistency in preventing, mitigating, and controlling general corrosion, SCC, and hydrogen embrittlement of AerMet 100 steel. (In this study, the CPC persistency is defined as a characteristic to resist detachment from the surface it protects or to remain in the environment in sufficient concentration to be protective. Its measure is the period of effective corrosion protection.)

EXPERIMENTAL PROCEDURE

SPECIMEN MATERIALS

Base Metal: AerMet 100 steel was selected as the base metal for specimen in this study. The chemical composition is shown in table 1.

Table 1: Chemical Composition of AerMet 100 Steel

Element	Weight (%)
C	0.23
Mn	0.03
Si	0.03
P	0.003
S	0.0009
Cr	3.03
Ni	11.09
Mo	1.18
Co	13.44
Cu	0.01
Fe	Balance

This steel was subjected to a heat treatment: preheating at 593°C (1,100°F) for 1.25 hr in an argon atmosphere, solution treating at 885°C (1,625°F) for 1.25 hr in an argon atmosphere, cooling in nitrogen atmosphere, freezing in dry-ice and alcohol of -73°C for 2 hr, and aging at 482°C (900°F) for 5 hr in air. This heat treatment resulted in the hardness of R_c 54 and the microstructure shown in figure A-1.

CPCs: Eighteen CPCs were employed for the testing. They were classified into three types according to the mechanism: solvent cutback (MIL-C-16173), water displacing (MIL-C-81309), and other. The solvent cutback CPCs were Esgard PL-4, Esgard PL-5, Nox-Rust 502, Tectyl 502C, Tectyl 511M, and Tectyl 894. The water displacing ones were Carwell AR500, Corrosion X, CRC3-36, and Omega 2775. The others were ACF-50, Boeshield T-9, Break Free SMX, Dinitol AV8, Fluid Film NAS, LPS2, ZC-010, and ZC-026. They could also be divided into two groups according to the fluidity, high and low. Those of high fluidity were ACF-50, Boeshield

T-9, Break Free SMX, Carwell AR500, Corrosion X, CRC 3-36, Dinitol AV8 Esgard PL-4, Esgard PL-5, LPS2, Nox-Rust 502, Tectyl 511M, ZC-010, and ZC-026. Those of low fluidity were Fluid Film NAS, Omega 2775, Tectyl 502C, and Tectyl 894.

OPEN CIRCUIT POTENTIAL MEASUREMENT

Open circuit potential (OCP) is an electrochemical parameter of corrosion resistance or susceptibility and measurable in a corrosion cell, consisting of a specimen electrode and a reference electrode (saturated calomel electrode (SCE)) in an aqueous electrolyte. In this study, the specimen electrode was a rectangular flat sheet of AerMet 100 steel, 1.5 in. long, 0.275 in. wide, and 0.04 in. thick, and its entire surface was coated with Stop-Off Lacquer, except an area of 0.20 x 0.275 in. on one face. Therefore, the working electrode had an area of 0.055 in.². The electrolyte was an aqueous 3.5% NaCl solution of pH 7.3. The specimen and reference electrodes were connected to the ground terminals of an electrometer, which read the OCP in reference to the SCE. The bare surface area of a specimen was initially polished, degreased with acetone and cleaned with distilled water. Subsequently, the area was coated with a CPC, cured for 1 hr at room temperature, and exposed to the electrolyte during OCP measurement in an apparatus, shown in figure A-2.

STRESS CORROSION CRACKING TEST

Since the cantilever bend and double cantilever beam SCC tests took a long time, an accelerated test method of rising step load (reference 22) was employed. The specimen was a square bar of 2.0 x 0.39 x 0.39 in. with a single edge 60 deg V-notch at the mid-length and L-T crack plane orientation, figure A-3. The specimen was fatigue-precracked to 1/2 thickness in air and then step-loaded in four-point bending under constant displacement control at room temperature, while held at a given potential in 3.5% NaCl solution of pH 7.3, in a test machine shown in figure A-4. The test machine, RSL 1000 SI-Multi-Mode System, included a bending frame, a tensile loading frame, an electrolyte reservoir, a pump for electrolyte circulation, a SCE, a platinum counterelectrode, a PC, and a printer. The load was increased at 2% of the ultimate bending strength each hour until the load dropped. The load drop corresponds to the threshold stress intensity for stress corrosion crack growth, K_{ISCC} . Its magnitude is quite important, because the threshold stress corrosion crack length varies as K_{ISCC}^2 , and calculated as a function of applied bending moment and crack length, using the following equation:

$$K_{ISCC} = \sigma \sqrt{\pi a} F(a/W)$$

where

K_{ISCC} : threshold stress intensity for stress corrosion cracking

σ : gross stress = $6M/bW^2$

M: bending moment = Px

P: applied load

x: moment arm length

b: specimen thickness

W: specimen width

a: crack length

$$F(a/W) = 1.122 - 1.40(a/W) + 7.33(a/W)^2 - 13.08(a/W)^3 + 14.0(a/W)^4$$

The precracked specimen was dipped in a CPC, filling the precrack and the notch-tip with the CPC, and cured for 1 hr at room temperature. Subsequently, the CPC coated specimen was subjected to the SCC test. During the test, the specimen was the working electrode in a three-electrode cell with a SCE and a platinum counterelectrode. OCP and electrode potential $V_{SCE} = -1.2$ V were applied to generate different amounts of hydrogen at the crack tip of a specimen under sustained bending. The K_{ISCC} values obtained at OCP and $V_{SCE} = -1.2$ V are the measures of resistance to SCC under free corrosion condition and hydrogen embrittlement, respectively.

RESULTS

OPEN CIRCUIT POTENTIAL

During its measurement, the OCP value of a specimen was initially shifted with time in the noble direction and subsequently stabilized. A typical example of the OCP variation with time is shown in figure A-5. The least electronegative or most noble one of the stabilized values was taken as the OCP of a specimen under a given condition. After a period of stabilization, the OCP value suddenly dropped or shifted in the active direction, indicating the loss of CPC effectiveness with time. The time from the start of test to the end of OCP stabilization was defined as the CPC effective period. The OCPs and CPC effective periods are listed with the CPC type and fluidity in table 2, and the former two are plotted in figure A-6 for the tested CPCs.

Table 2: OCP and CPC Effective Period

CPC	OCP (V)	Effective Period (hr)	Type MIL-C-	Fluidity
No Coating	-0.510	-	-	-
Nox-Rust 502	-0.190	23.3	16173 ⁽¹⁾	high
Boeshield T-9	-0.189	21.2	-	-
Carwell AR500	-0.168	21.7	81309 ⁽²⁾	high
Dinitol AV8	-0.143	7.8	-	high
Break Free SMX	-0.130	6.3	-	high
ZC-026	-0.085	7.2	-	high
Esgard PL-4	-0.066	21.8	16173	high
Tectyl 511M	-0.053	7.8	16173	high
Tectyl 894	-0.050	19.5	16173	low
Esgard PL-5	-0.040	> 24	16173	high
Corrosion X	-0.038	> 24	81309	high
ACF-50	-0.035	18.0	-	high
Fluid Film NAS	-0.005	21.1	-	low
Tectyl 502C	-0.030	> 24	16173	low
CRC 3-36	-0.020	19.8	81309	high
Omega 2775	-0.020	> 24	81309	low
LPS2	-0.008	> 24	-	high
ZC-010	+0.030	> 24	-	high

NOTES: (1) MIL-C-16173: CPCs, Solvent Cutback, Cold-Application.

(2) MIL-C-81309: CPCs, Water Displacing Ultra-Thin Film.

RESISTANCE TO STRESS CORROSION CRACKING AND HYDROGEN EMBRITTLEMENT

The K_{ISCC} values, determined at the OCP (-0.51 V) of the bare specimen and $V_{SCE} = -1.2$ V, are listed in table 3 and plotted in figure A-7 for the specimens bare and coated with CPC.

Table 3: K_{ISCC} Values of Uncoated and CPC Coated Specimens

CPC	K_{ISCC} (ksi $\sqrt{\text{in.}}$)		Type MIL-C-	Fluidity
	OCP	$V_{SCE} = -1.2 \text{ V}$		
No Coating	23.3	14.0	-	-
Tectyl 511M	82.7	35.6	16173 ⁽¹⁾	high
Dinitol AV8	86.3	40.7	-	high
Boeshield T-9	96.2	43.1	-	high
CRC 3-36	103.9	33.5	81309 ⁽²⁾	high
ACF-50	104.6	53.3	-	high
LPS2	104.6	40.6	-	high
Esgard PL-4	104.9	61.7	16173	high
ZC-026	105.6	88.7	-	high
Nox-Rust 502	106.2	84.2	16173	high
Tectyl 894	106.6	42.2	16173	low
Tectyl 502C	106.9	76.2	16173	low
Break Free SMX	107.1	106.8	-	high
Fluid Film NAS	107.4	91.4	-	low
Esgard PL-5	107.5	86.8	16173	high
Corrosion X	107.6	33.4	81309	high
ZC-010	108.7	40.1	-	high
Carwell AR500	108.9	87.3	81309	high
Omega 2775	108.9	79.8	81309	low

NOTES: (1) MIL-C-16173: CPCs, Solvent Cutback, Cold-Application.

(2) MIL-C-81309: CPCs, Water Displacing Ultra-Thin Film.

DISCUSSION

PREVENTION/MITIGATION OF GENERAL CORROSION

It has been known that the more negative the value of OCP, the greater the tendency for or susceptibility to general corrosion. The largest negative value of OCP, -0.51 V, for the bare AerMet 100 steel indicates the most pronounced tendency for general corrosion in 3.5% NaCl solution. On the other hand, with CPC coating, the OCP value became less negative or it was shifted in the noble direction, evidencing prevention/mitigation of general corrosion. From table 2, it is clear that CPC ZC-010, having a positive value of OCP, is most effective in general corrosion prevention among the CPCs tested. The CPC effective period is longest for the CPC ZC-010 and shortest for CPC Break Free SMX in 3.5% NaCl solution, as shown in table 2. Mostly, a less negative or a positive OCP has a longer CPC effective period, indicating more persistent.

The effect of CPC fluidity on general corrosion is not so clear from the results obtained. Some CPCs of lower fluidity seem to be more effective and persistent in prevention/mitigation of general corrosion.

PREVENTION/MITIGATION OF HYDROGEN EMBRITTLEMENT AND STRESS CORROSION CRACKING

The hydrogen generation at a crack tip is highly accelerated at the electrode potential -1.2 V, compared to that at the OCP, in 3.5% NaCl solution. Therefore, the values of K_{ISCC} at -1.2 V and the OCP have been taken as the measures of susceptibility to hydrogen embrittlement and SCC, respectively.

The K_{ISCC} values were observed to be lower at $V_{SCE} = -1.2$ V than at the OCP for the bare and CPC coated specimens, as shown in table 3 and figure A-7. Furthermore, they increased with CPC coating 3.6 ~ 4.5 times at the OCP and 2.4 ~ 6.5 times at $V_{SCE} = -1.2$ V, indicating the CPC effectiveness in the prevention/mitigation of SCC and hydrogen embrittlement. Carwell AR500 and Omega 2775 were most effective in prevention/mitigation of SCC and Break Free SMX most effective in that of hydrogen embrittlement.

The effect of CPC fluidity on K_{ISCC} at OCP and $V_{SCE} = -1.2$ V is not so clearly definable with the result obtained. Mostly, the CPC of lower fluidity tends to induce a greater value of K_{ISCC} at OCP or greater effectiveness in prevention/mitigation of SCC.

CONCLUSIONS

The tested 18 CPCs are effective in prevention/mitigation of general corrosion, SCC, and hydrogen embrittlement of AerMet 100 steel in 3.5% NaCl solution. However, there is a difference, large or small, in the effectiveness and persistency among those CPCs.

CPC ZC-010 is most effective and persistent in prevention/mitigation of general corrosion in 3.5% NaCl solution. This CPC is also good for the prevention/mitigation of SCC in 3.5% NaCl solution.

Carwell AR500 and Omega 2775 are most effective in prevention/mitigation of SCC and Break Free SMX most effective in that of hydrogen embrittlement in 3.5% NaCl solution.

RECOMMENDATIONS

Shipboard determination of the long term CPC effective period for AerMet 100 steel in service environment. (In this investigation, only the short term CPC effective period in 3.5% NaCl solution was determined.)

Investigation of CPC for corrosion prevention/mitigation of the other landing gear steels, such as 300M and 4340.

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LIST OF ABBREVIATIONS AND FORMULAS

CPC	Corrosion Preventive Compound
IGC	Intergranular Corrosion
OCP	Open Circuit Potential
SCC	Stress Corrosion Cracking
SCE	Saturated Calomel Electrode
UTS	Ultimate Tensile Strength
K_{IC}	Plane Strain Fracture Toughness
K_{ISCC}	Threshold Stress Intensity for Stress Corrosion Cracking
V_{SCE}	Electrode Potential versus Saturated Calomel Electrode

APPENDIX A
FIGURES

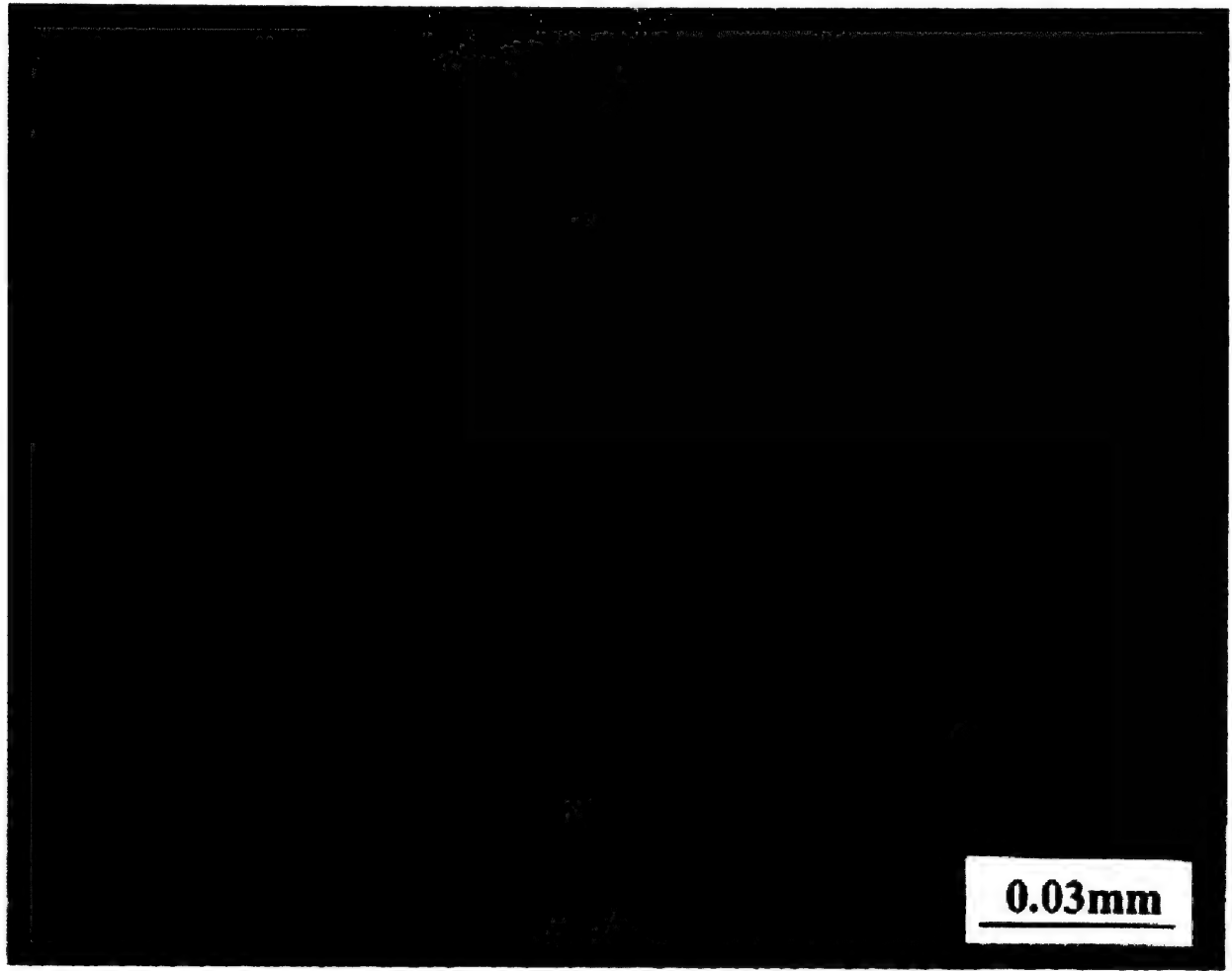


Figure A-1: Microstructure of AerMet 100 Steel

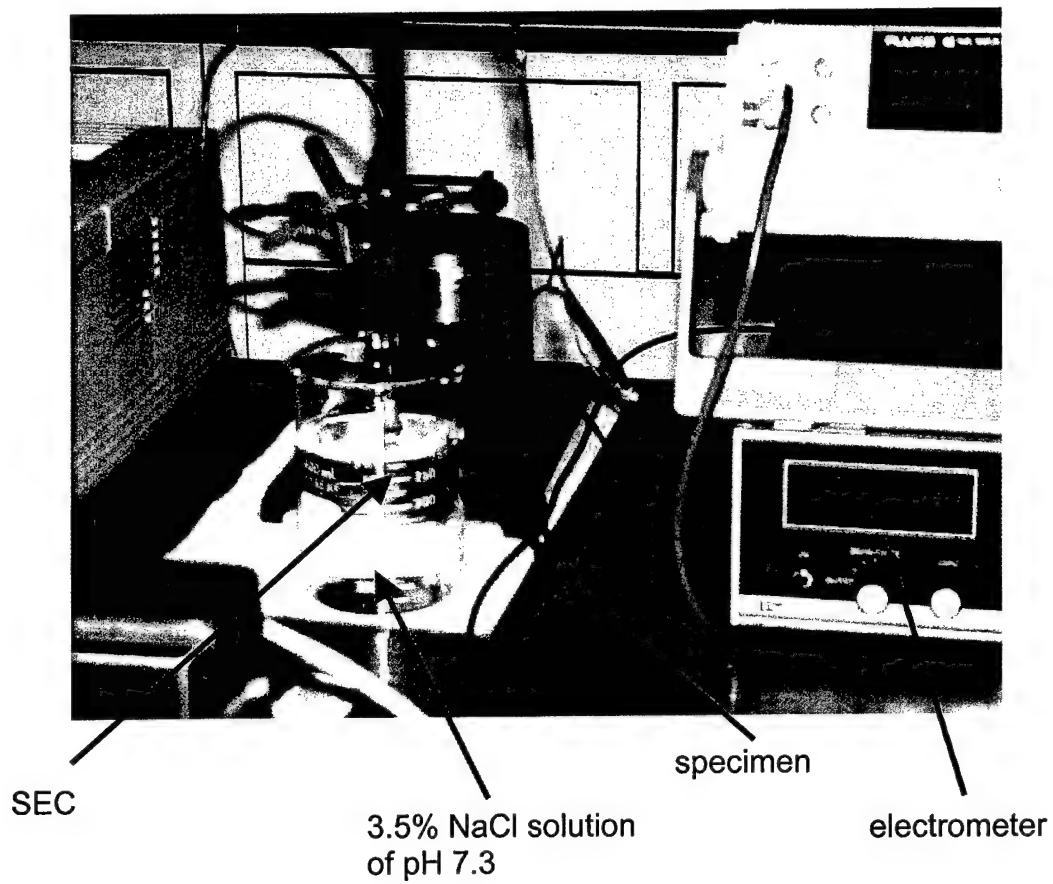
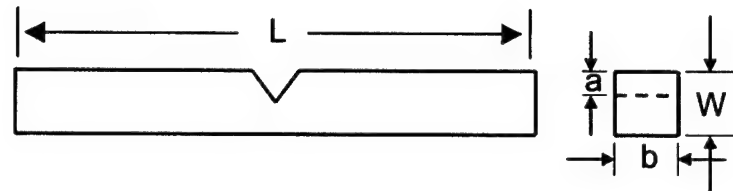


Figure A-2: Apparatus for OCP Measurement



$L = 2.0$ in.
 $W = b = 0.39$ in.
 $a = 0.195$ in.

Figure A-3: Square Bar Specimen with Single Edge V-Notch

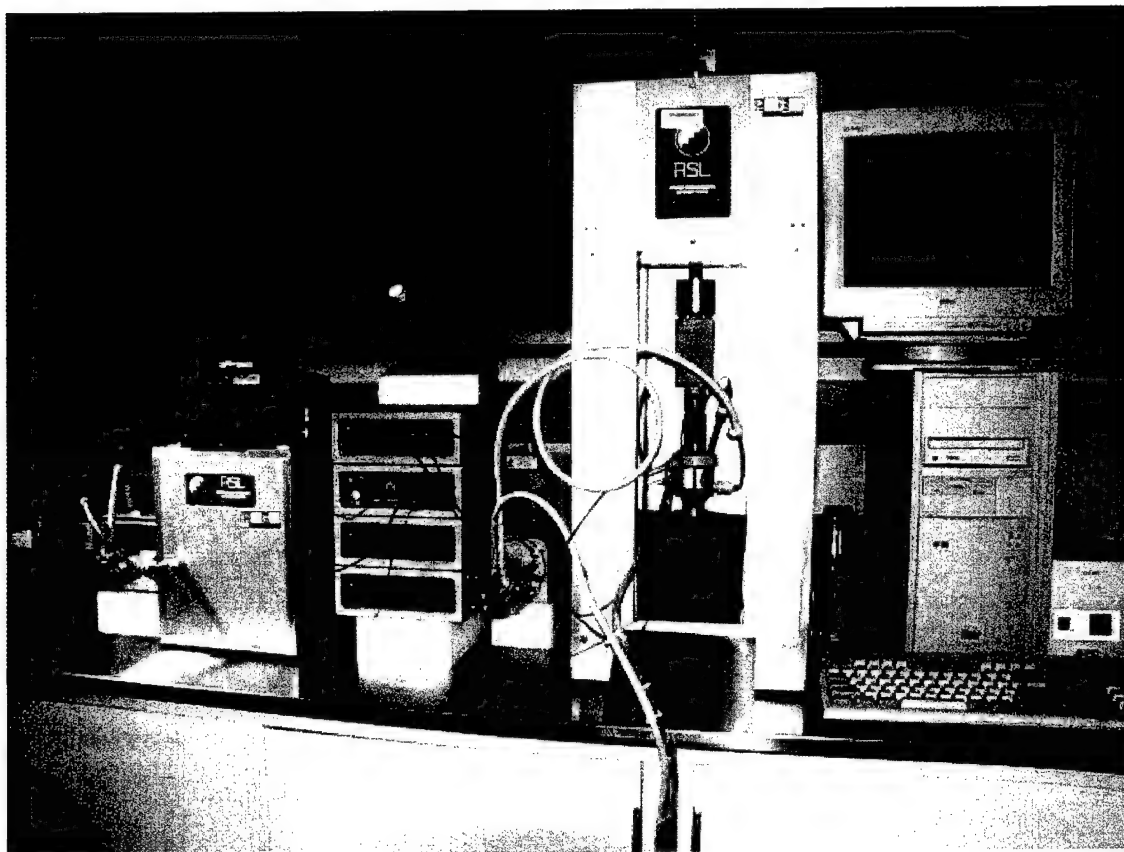


Figure A-4: Rising Step Load Test Machine

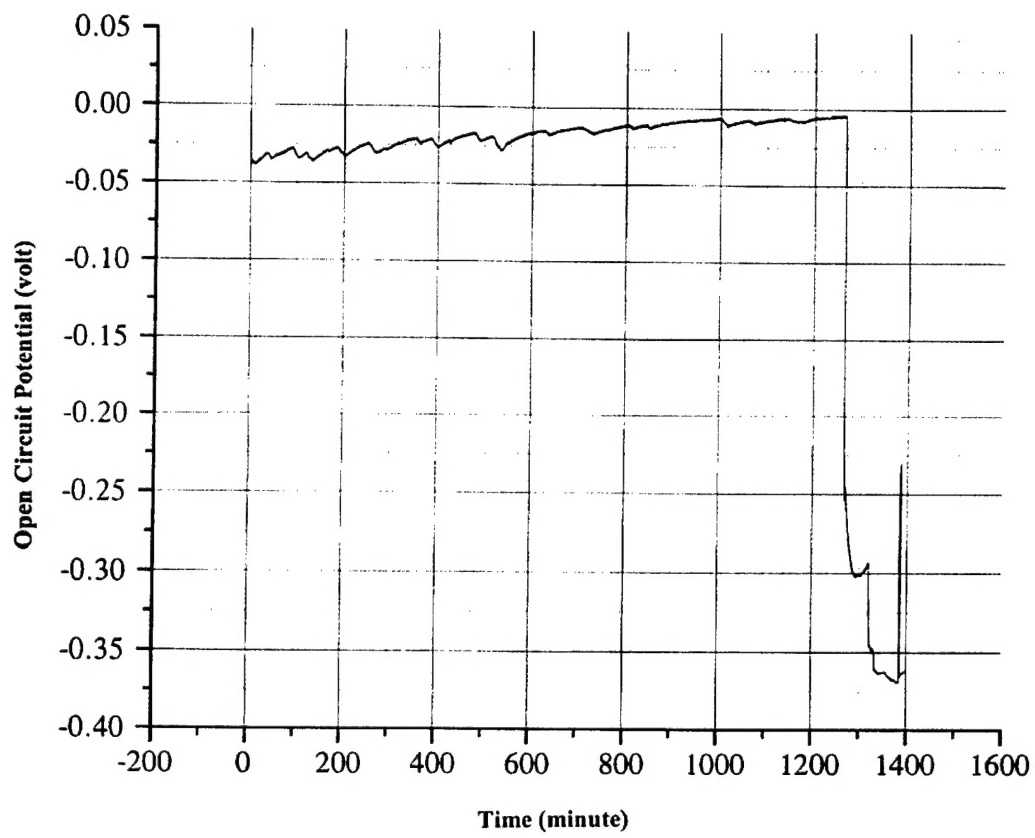


Figure A-5: Typical Example of Variation of OCP with Time

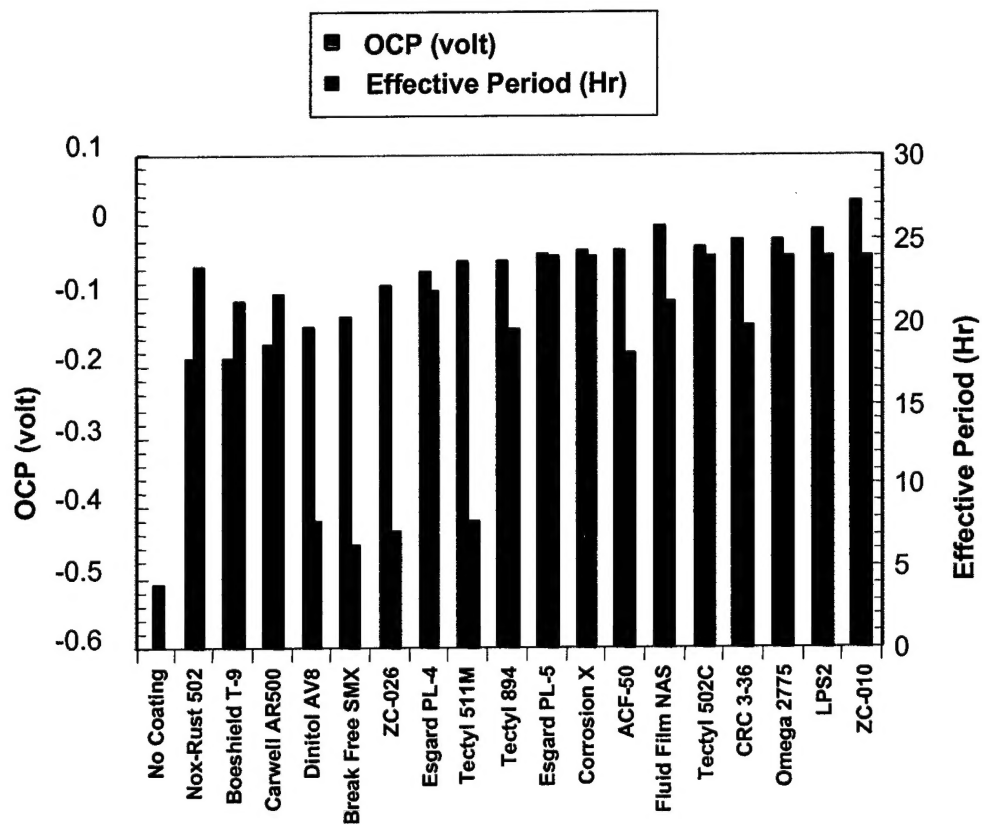


Figure A-6: OCPs and Their Effective Periods

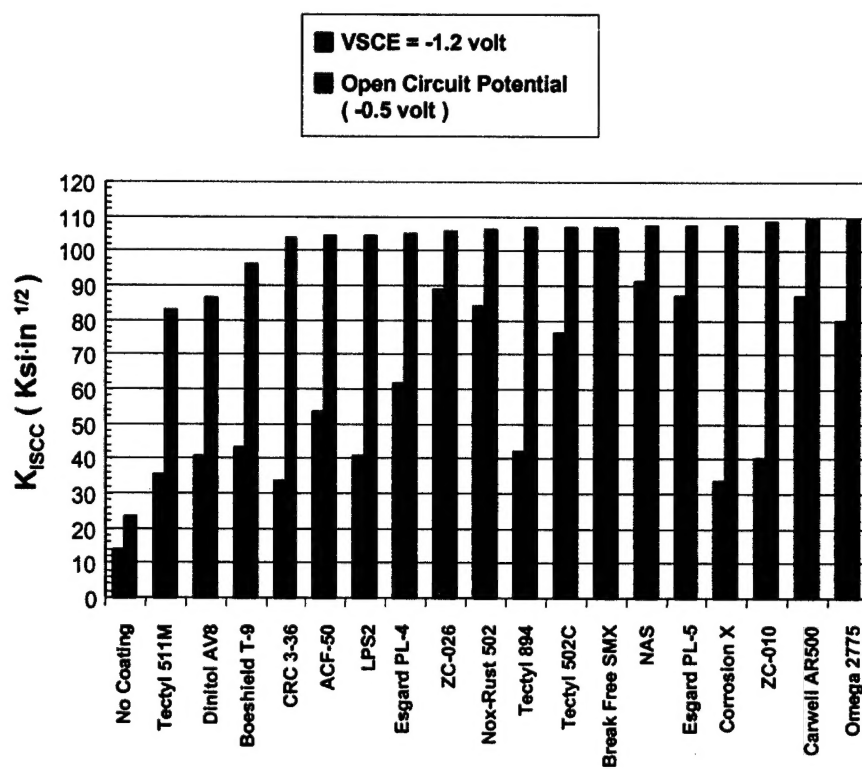


Figure A-7: K_{ISCC} Values at $V_{SCE} = -1.2$ V and OCP

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